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Mazdoor Kisan Shakti Sangathan

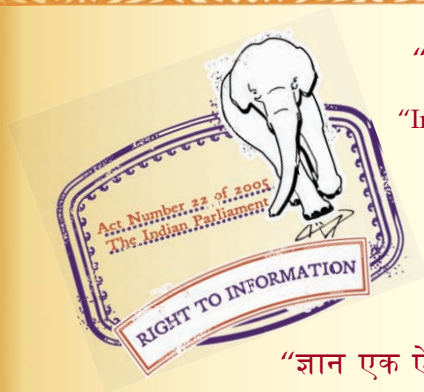
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IS 11126 (1984): 3-toluidine [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard
SPECIFICATION FOR
3-TOLUIDINE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR 3-TOLUIDINE

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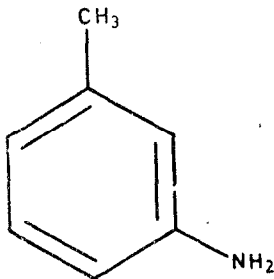
Indian Standard

SPECIFICATION FOR 3-TOLUIDINE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 December 1984, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 3-Toluidine (C_7H_9N) is used as an intermediate in the manufacture of coal tar dyestuffs and other chemicals. It is also known as *m*-toluidine or 3-aminotoluene. It is represented by the following structural formula :



3-TOLUIDINE

(MOLECULAR MASS 107.2)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified values in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 3-toluidine.

*Rules for rounding off numerical values (revised).

2. REQUIREMENTS

2.1 Description — The material shall be pale-brown or reddish-brown liquid, free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3-TOLUIDINE

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay (by diazotization), percent by mass, <i>min</i>	99	A-1
ii)	Distillation range, °C	99 percent will distill within 200 to 203°C	A-2
iii)	Solubility in hydrochloric acid solution	To pass the test	A-3
iv)	Total isomers, percent by mass, <i>max</i>	1	A-4

3. PACKING AND MARKING

3.1 Packing — The material shall be suitably packed in steel drums lined with polyethylene bags or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — The containers shall be marked with the following:

- a) Name of the material;
- b) Tare, gross and net mass of the material;
- c) Name of the manufacturer and his recognized trade-mark, if any;
- d) Batch No.; and
- e) Date of manufacture.

3.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under:

‘DANGER OF CYANOSIS; BLOOD POISON, HAZARDOUS LIQUID AND VAPOUR RAPIDLY ABSORBED THROUGH SKIN’.

3.2.2 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI-marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Assay of 3-toluidine shall be determined on each of the individual sample.

4.2.2 Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Test shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(Table 1 and Clause 5.1)

METHODS OF TEST FOR 3-TOLUIDINE

A-1. ASSAY (BY DIAZOTIZATION)

A-1.1 Preparation of Sample for Test — Transfer about 100 ml of the material into a stoppered flask containing anhydrous calcium chloride or anhydrous sodium sulphate. Shake well for about 15 minutes. Decant the liquid through a dry filter paper to a dry flask. Use this *prepared sample* for the determination of distillation range and assay.

A-1.2 Reagents

A-1.2.1 Hydrochloric Acid — see IS : 265-1976*.

A-1.2.2 Sodium Nitrite Solution — 0.2 N.

A-1.2.3 Potassium Bromide

A-1.2.4 Starch Iodide Test Papers

A-1.3 Procedure

A-1.3.1 Weigh 4.5 to 5 g sample in a previously weighed and dried stoppered weighing bottle. Transfer the sample to a 250 ml volumetric flask. Add 3 ml of concentrated hydrochloric acid and transfer it to the flask. Wash the weighing bottle with water. Repeat the washing of bottle thoroughly with 3 ml of concentrated hydrochloric acid, and water. Transfer these washings to the flask each time. Add 10 ml more of concentrated hydrochloric acid, then stir well to dissolve the sample completely. Dilute solutions to the mark with distilled water and shake well.

A-1.3.2 Take 50 ml vacupet and rinse twice with solution from volumetric flask. Withdraw 50 ml of solution from flask to a one-litre beaker. Add 400 ml of water, 30 ml of concentrated hydrochloric acid and ice to cool the solution between 0 to 5°C. Titrate against standard sodium nitrite solution from burette after adding 3 g of potassium bromide. End point is bluish-violet ring developing on starch iodide paper with one drop of solution from beaker. The bluish-violet ring should persist for at least 2 minutes which is confirmed after 2 minutes on starch iodide paper. Note the reading.

*Specification for hydrochloric acid (*second revision*).

A-1.3.3 Repeat the experiment of assay by diazotization. The two titration readings should not vary by more than 0.1 ml.

A-1.4 Calculation

$$\text{Assay (by diazotization) , percent by mass} = \frac{V \times N \times 10.72}{M}$$

where

V = volume in ml of sodium nitrite solution used,

N = normality of sodium nitrite solution,

M = mass in g of the sample aliquot taken for test.

A-2. DETERMINATION OF DISTILLATION RANGE

A-2.1 Carry out the distillation range of the sample in accordance with 6 of IS : 5299-1969*.

A-3. DETERMINATION OF SOLUBILITY IN HYDROCHLORIC ACID

A-3.1 Carry out the test in accordance with 10 of IS : 5299-1969*. There shall not be more than a slight opalescence in the solution.

A-4. DETERMINATION OF TOTAL ISOMERS

A-4.0 General — The estimation of total isomers is done by thin layer chromatography.

A-4.1 Apparatus

A-4.1.1 Thin Layer Chromatographic Plate — glass plate of size 20 × 20 cm, coated uniformly with silica gel-G of 0.5 mm thickness and activated at 110°C for 1 h.

A-4.1.2 Micropipette

A-4.1.3 Developing Chamber

A-4.1.4 Chromatographic Sprayer

A-4.2 Reagents

A-4.2.1 2-Toluidine — pure.

A-4.2.2 4-Toluidine — pure.

*Methods of sampling and tests for dye intermediates.

A-4.2.3 Developing Solvent — benzene / hexane / methanol / acetic acid (80:16:5:5).

A-4.2.4 Spray Solution — 1 percent *p*-diaminobenzaldehyde and 10 percent stannous chloride (1:1).

A-4.3 Procedure

A-4.3.1 Weigh accurately 1.0 g of the sample and dissolve it in 50 ml of a mixture of water and methanol (1:1) by warming on a water-bath, cool, transfer to a 100-ml volumetric flask and dilute to 100 ml with the water-methanol mixture (1:1).

A-4.3.2 Weigh accurately 0.1 g of the 2-toluidine and 4-toluidine separately and dissolve it in 100 ml flasks with the mixture of water and methanol as in A-4.3.1. Take 10-ml of these solutions in the flasks and dilute to 100 ml with the water-methanol mixture (1:1).

A-4.3.3 Spot 10 μ l of the sample (A-4.3.1) and the reference solution (A-4.3.2) on the thin layer plate. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in the ascending manner for about 80 to 100 minutes. Remove the plate from the chamber and dry off the solvent in an air drier. Spray the plate with spray solution (A-4.2.4) and heat the plate at 100°C. After a few minutes examine visually the intensity of colour of each zone developed with the sample solution and compare it with that developed with the reference solution.

AMENDMENT NO. 1 MAY 2002
TO
IS 11126 : 1984 SPECIFICATION FOR 3-TOLUIDINE

(*Page 3, clause 0.2, Structural Formula*) — Insert the following below the structural formula:

‘(CAS No. 108-44-1).’

[*Page 4, Table 1, Sl No. (iii)*] — Substitute the following for the existing:

(1)	(2)	(3)	(4)
iii)	Matter insoluble in dilute hydrochloric acid, percent by mass, <i>Max</i>	0.2	when tested as per IS 5299 : 2001

(PCD 11)

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